

AFFIDAVIT**RE: Japanese Patent Laid-Open Number H4-190780**

I, Yoshinori Tanabe, Ark Mori Bldg, 28F, No.12-32,
Akasaka 1-chome, Minato-ku, Tokyo, Japan, do duly swear and
depose:

THAT I am a graduate from Waseda University, school of
Science and Technology, Department of Applied Chemistry,
Course of Chemical Engineering.

THAT I am presently employed by NBT Corporation of Tokyo,
Japan and have been so employed since April 1997, during which
I have acted as a Section Manager in the Operations Department
of said Company for the preparation of translation for the
Japanese courts and the patent offices, including the United
States Patent and Trademark Office.

THAT I understand the Japanese and English languages and
the attached English document is believed to be a full, true
and faithful translation of the Japanese Patent Laid-Open
Number H4-190780 published July 9, 1992.

THAT I declare further that all statements made herein
of my knowledge are true and that all statements made on
information and belief are believed to be true; and further
that these statements were made with the knowledge that
willful false statements and the like so made are punishable
by fine or imprisonment, or both, under Section 1001 of Title
18 of the United States Code.

March 20, 2002


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Japanese Patent Laid-Open No. 190,780/1992

Laid-Open Date: July 9, 1992

Application Number: 324,931/1990

Application Date: November 26, 1990

METHOD FOR THE MANUFACTURE OF VINEGAR USING CITRUS FRUITS
AS MATERIALS AND A PRODUCT THEREOF

What is Claimed is:

1. A method for the manufacture of vinegar using citrus fruits as materials, characterized in that, citrus fruit juice is treated with an enzymatic preparation to give a clear fruit juice, acidity of the fruit juice is adjusted, the juice is sterilized by heating and cooled, alcohol is added to the said clear fruit juice, acetic acid bacteria are inoculated followed by subjecting to an acetic acid fermentation to manufacture acetic acid, the mixture is aged, waste bacteria in the citrus fruit vinegar are filtered off using a filter and fruit juice obtained from unripe citrus fruits is added for flavoring to give a product.

2. Vinegar which is a citrus fruit vinegar manufactured by a method for the manufacture of vinegar using citrus fruits as materials, characterized in that,

citrus fruit juice is treated with an enzymatic preparation to give a clear fruit juice, acidity of the fruit juice is adjusted, the juice is sterilized by heating and cooled, alcohol is added to the said clear fruit juice, acetic acid bacteria are inoculated followed by subjecting to an acetic acid fermentation to manufacture acetic acid, the mixture is aged, waste bacteria in the citrus fruit vinegar are filtered off using a filter and fruit juice obtained from unripe citrus fruits is added for flavoring to give a product.

3. The method for the manufacture of vinegar manufactured from the citrus fruits according to claim 1 or 2 as the materials and a vinegar product thereof, wherein the technical characteristic is that the fruit juice containing 100% of citrus fruit juice is treated with pectinase as an enzymatic preparation to give a clear fruit juice.

Detailed Description of the Invention:

[Technical Field of the Invention]

The method for the manufacture of vinegar manufactured from citrus fruits as the materials and the product thereof in accordance with the present invention are to provide a method for the manufacture of vinegar manufactured by a fruit juice containing 100% of citrus

fruits such as mandarin orange, navel orange, ponkan orange, unshu mandarin orange and hassaku mandarin orange as materials for vinegar, a vinegar product which is a citrus vinegar manufactured by that method and a product concerning application of using the said product.

[Prior Art]

The conventional steps for the manufacture of vinegar are that fruit juice which is a material is sterilized, alcohol is added thereto, acetic acid bacteria are inoculated followed by subjecting to acetic acid fermentation and the mixture is aged, filtered and sterilized to give a product.

When the conventional method is applied to citrus fruits and vinegar is manufactured from citrus fruit juice as a material, a technique where fibrous matters which are the cause for color change are completely removed is necessary. Since such a technique is not available in the conventional manufacturing steps, it is difficult in view of technique whereby, when vinegar is manufactured from a material comprising 100% of natural fruit juice, color of the vinegar changes and its flavor is lost. Due to such a reason, the earnest efforts for research and development of a technique for the manufacture of vinegar from a material comprising

100% of citrus fruit juice have been still unable to be practically utilized for solving the unsolved problems by which vinegar is manufactured and sold as a commercial product.

[Problems to be Solved by the Invention]

When vinegar is manufactured in the prior art from fruit juice of about 30% of fruit juice content, changes in color do not take place too much and the product after aging and filtration is without a reduction in transparency of vinegar and some fragrance still remains therein. However, when vinegar is manufactured from a 100% fruit juice, many fibrous matters contained in the fruit juice change to brown pigments (this is called browning) in about the third month during the aging of vinegar. Even when they are filtered off, it is not possible to completely remove the brown pigments whereupon the vinegar is turbid in brown color and the flavor of vinegar is completely lost. Thus, it is not possible to give a satisfactory product which meets with the requirements for manufacturing and selling as citrus fruit vinegar.

The above-mentioned problems are the problems which have been left unsolved in the manufacture of vinegar. When a novel technique where vinegar can be manufactured from a 100% fruit juice by creation of a

unique technical idea utilizing natural law and rules is practically applied, it will be possible that citrus fruits which are abundantly produced in this country are squeezed and the fresh fruit juice is made into citrus fruit vinegar. Therefore, development of such a technique is a matter by which some economical effects can be expected but, since a technical resolution for the unsolved matters is necessary, that has not been solved yet.

As a result of the novel technique of the present invention, both technical problems of browning reaction and loss of flavor are now solved. Therefore, a method for the manufacture of vinegar containing clear and fresh flavor components can be achieved from a fruit juice comprising 100% of citrus fruit juice whereby it is now possible to practically utilize a technique for the manufacture of vinegar having transparency, flavor and good taste from the said 100% fruit juice.

[Means for Solving the Technical Problems of the Invention]

A means for solving the technical problems concerning the method for the manufacture of vinegar from citrus fruit as a material and a product prepared thereby according to the present invention is that fibrous matters contained in fruit juice are treated with

pectinase which is an enzymatic preparation so that pectin of pulp is hydrolyzed to make its low molecule whereupon a clear fruit juice in which the citrus fruit juice is made smooth is prepared and then fruit juice obtained by squeezing of unripe citrus fruit is added to citrus vinegar prepared by processing the above-prepared clear fruit juice, flavor is given thereto whereupon the conventional problems which have been technically left unsolved have now been solved.

Now the said manufacturing method and the manufacturing steps for vinegar manufactured by said means from citrus fruit juice for solving the technical problems in the product will be illustrated in detail.

Fruit juice comprising 100% of citrus fruit juice is treated with pectinase, an enzymatic preparation, to give a clear fruit juice.

To the clear fruit juice is added sodium citrate so as to adjust the pH to 4.6, the mixture is sterilized by heating at 60°C for 30 minutes and cooled, ethyl alcohol is added thereto to make its concentration 6%, the mixture is transferred to a fermentation tank for an acetic acid fermentation, acetic acid bacteria are inoculated and fermentation is carried at 25-30°C out until the acetic acid concentration reaches 5.0%.

When the acetic acid concentration of the fruit

juice in a fermentation tank reaches 5.0%, it is aged in a cool and dark place at not higher than 25°C for five to six months. The waste bacteria in the citrus fruit vinegar resulted from the aging is filtered off using a filter and then a 100% fruit juice obtained by squeezing of unripe citrus fruits is added to give a flavor whereupon a product is prepared.

The resulting vinegar which is citrus fruit vinegar is filled in containers, followed by sealing, sterilized by heating at 60°C for 30 minutes and cooled with cool water to give a final product.

[Function]

As a result of action of pectinase which is an enzymatic preparation added to citrus fruit juice, pectin which is a fibrous matter is hydrolyzed to make its low molecule whereby a clear fruit juice in a smooth state is prepared. If a 100% fruit juice obtained by squeezing of citrus fruit is made into a clear fruit juice as such by treating with pectinase which is an enzymatic preparation, it is now possible to produce vinegar without a big browning reaction when the adjusted fruit juice is transferred to a fermentation tank, acetic acid bacteria are inoculated thereto, an acetic acid fermentation is carried out until the acetic acid concentration reaches 5% and aging is carried out in a

cool and dark place for five to six months. On the contrary, when a fruit juice material comprising 100% of citrus fruit juice which has not been treated with an enzymatic preparation is adjusted to pH 4.6, acetic acid bacteria are inoculated, acetic acid fermentation is completed and aged, then the result is that, after three months, browning reaction takes place and flavor is lost. Even when the vinegar as such is filtered using a filter, it is still impossible to completely remove the brown color.

Accordingly, the action of pectinase which is an enzymatic preparation is that the fruit juice as a material is treated with pectinase to give a clear fruit juice whereby browning reaction during the manufacturing steps of from citrus fruit juice to vinegar does not occur.

Vinegar which is prepared by aging for five to six months in a cool and dark place at not higher than 25°C is treated with a filtering agent and an activated carbon so that the waste bacteria in vinegar are adsorbed therewith and removed and then a 100% fruit juice obtained by squeezing of unripe citrus fruit is added thereto whereby a flavor is given. The fruit juice obtained from unripe fruit is added for giving a flavor to the resulting vinegar.

A fruit juice obtained by squeezing of ripe citrus fruit has a high saccharide concentration and, as a result, vinegar having a high saccharide concentration is produced. When ripe citrus fruit is used, its fruit juice gives vinegar having a high saccharide concentration via the steps for the manufacture of vinegar and, when an enzyme preparation is applied to the citrus fruit juice which is a material during the manufacturing steps, the material becomes a clear fruit juice whereby the change into brown color which takes place during the aging of vinegar does not occur too much. Even if that occurs to some extent, brown pigments are completely adsorbed with activated carbon which is a filtering material as well as an adsorbent when filtered through a filter and, in the final stage, the vinegar is a colorless and transparent liquid.

It is difficult that all of the natural flavors contained in citrus fruit juice are maintained during the steps from the starting fruit juice to the final vinegar. Therefore, during its manufacturing steps, an enzymatic preparation is applied whereby vinegar which does not cause browning is able to be manufactured from the resulting clear fruit juice while, in order to supplement the flavor components which are lost during the manufacturing steps, a fruit juice obtained by

squeezing of unripe fruit is added after filtering the product whereby the citrus fruit vinegar is flavored to give the final product.

The vinegar which is manufactured by the method of the present invention is colorless and transparent where turbidity caused by browning does not take place at all and, since it contains much saccharide components, has sweetness and contains extract substances, the said vinegar is mild to tongue and has a good taste. Characteristics of the nutrient components of this vinegar are that saccharides, extracts, vitamin C, etc. are abundantly contained therein.

[Examples]

The attached drawing is to illustrate a method for the manufacture of vinegar using citrus fruit as a material according to the present invention and the manufacturing steps for the product by way of a flow chart.

Now there will be illustrated an example for the steps for the manufacture of citrus fruit vinegar as fruit vinegar from unshu mandarin orange utilizing the manufacturing method of the present invention and, at the same time, there will be illustrated in detail a mandarin orange vinegar which is a citrus fruit vinegar manufactured as a fruit vinegar by way of an example of

the manufacturing method.

Unshu mandarin orange which is a material is squeezed to manufacture a fruit juice comprising 100% of citrus fruit. The fruit juice is stored in a freezer so that it can be taken out and used if and when necessary. In order to make the thawed fruit juice clear, pectinase which is an enzymatic preparation is applied to fibrous matter precipitated in the fruit juice so that pectin is hydrolyzed to make the fruit juice in a smooth state whereupon a clear fruit juice is prepared.

In order to adjust the acidity of the resulting clear mandarin orange fruit juice, sodium citrate is added to make its pH 4.6.

The mandarin orange fruit juice adjusted as such is sterilized by heating at 60°C for 30 minutes and cooled and ethyl alcohol is added to make its concentration to the adjusted mandarin orange fruit juice 6%. Moromi (unrefined Japanese sake) may be added instead of ethyl alcohol. When acetic acid bacteria are inoculated to the fruit juice and fermentation is carried out, alcohol changes to acid.

The fruit juice prepared as such is transferred to a fermentation tank, acetic acid bacteria are inoculated, acetic acid fermentation is carried out at 25-30°C and, when the acetic acid concentration reaches 5.0%, the

fermentation is stopped. When temperature of the fermentation tank is 30°C or higher, rate of browning becomes high and, therefore, the temperature of 25-30°C is appropriate. It is important to take care that the temperature does not rise higher than above.

The acetic acid fermentation is finished and then aging is carried out in a cool and dark place of 25°C or lower for five to six months. During the aging period, considerably dark coloration is resulted and that can be removed by adsorbing with active carbon.

In order to remove the waste bacteria in the mandarin orange vinegar prepared as such by adsorbing with active carbon, the said vinegar is filtered through a filter equipped with active carbon acting as a filtering material and also as an adsorbing agent.

A 100% fruit juice obtained by squeezing of unripe fruit of unshu mandarin orange is added thereto to give flavor to finish.

The finished mandarin orange vinegar is filled in containers, sterilized by heating at 60°C for 30 minutes and cooled with cold water to give the final product. This will be called mandarin orange vinegar 100.

The nutrients contained in mandarin orange vinegar and in commercially available apple vinegar and rice vinegar (both will be referred to as commercially-

available vinegar) are compared and shown in Table 1.

Table 1

(unit: mg/100 g except sugar concentration and pH)

	Mandarin Orange Vinegar 100	Apple Vinegar	Rice Vinegar
Saccharide Concentration	10.1%	4.2%	3.7%
Extract Substances	8.80	5.89	4.00
Ashes	0.7	0.2	0.64
Calcium	2.5	2.0	2.0
Potassium	45.5	55.0	6.0
Vitamin C	18.0	0	0
pH	3.80	3.10	2.5

Fruit juice content of the mandarin orange vinegar 100 is 100%. Fruit vinegar in general meets with the requirements of JAS (Japanese Agricultural Standard) concerning foods when the fruit juice content is 30% or more.

As shown in Table 1, saccharide concentration, extract substances amount and vitamin C amount in the mandarin orange vinegar 100 are 10.1%, 8.80 mg and 18.0 mg and they are higher than the data for the commercially available vinegars. Mandarin orange vinegar 100 contains much saccharides and has a sweet taste. Because the amino acids which are extract substances are abundant, its mouth-feel is smooth and taste is good. There are much inorganic substances such as calcium and potassium. A lot of vitamin C is contained. Due to such

characteristics, mandarin orange vinegar 100 is able to be practically used as fruit vinegar.

This mandarin orange vinegar 100 has a fresh orange flavor, non-vicious sweetness and acid taste and can be used in various meals. Examples of the products where the mandarin orange vinegar 100 is used are vinegar for *chiri* (fish stew seasoned with bitter-orange juice), *sumiso* (vinegar mixed with soybean paste), *sujoyu* (soy sauce made sour with vinegar), *oroshisujoyu* (soy sauce made sour with vinegar assorted with grated radish), *shogasujoyu* (soy sauce made sour with vinegar assorted with ginger), *gomasu* (vinegar mixed with sesame), *awasezu* (compounded vinegar), vinegar *à la chinoise*, *amazusosu* (Worcester sauce mixed with sweet vinegar), marinade, dressing, healthy drink made from honey and mandarin orange vinegar and fruit jelly mixed with mandarin orange vinegar.

Examples of the compounding of mandarin orange vinegar drink (Application Example 1) and mandarin orange vinegar jelly (Application Example 2) are illustrated in Tables 2 and 3.

Table 2

Compounding Example of Mandarin Orange Vinegar Drink

Materials Used	Compounding (in %)
Mandarin Orange Vinegar	10
Honey	5
Oligosaccharides	5
Vitamin C	0.01
Vitamin B ₂	0.001
Water	79.989
Total	100

Table 3

Compounding Example of Mandarin Orange Vinegar Jelly

Materials Used	Compounding (in %)
Mandarin Orange Vinegar	10
Sugar	15
Honey	5
Oligosaccharides	3
Jelly Material	1
Fragrance	0.1
Water	65.9
Total	100

[Advantages]

Method for the manufacture of vinegar prepared from citrus fruits as a starting material in accordance with the present invention and the product are characterized in that a 100% citrus fruit juice is treated with an enzymatic preparation (pectinase) to give a clear fruit juice, alcohol is added thereto, acetic acid bacteria are inoculated, acetic acid fermentation is carried out to produce acetic acid, the mixture is aged and, for

giving a flavor, fruit juice obtained from unripe citrus fruit is added to give a product. Thus it is now possible to manufacture transparent vinegar which is a citrus fruit vinegar having both good flavor and taste from a 100% fruit juice obtained by squeezing of citrus fruits such as mandarin orange, navel orange, ponkan orange, unshu mandarin orange and hassaku mandarin orange. The resulting product contains a lot of saccharides, extract substances and vitamin C and, since application examples of the product are in broad range, a big economical effect can be greatly expected when this technique is practically utilized at an early date.

Brief Explanation of the Drawing:

The attached drawing is to illustrate a method for the manufacture of vinegar using citrus fruit as a material according to the present invention and the manufacturing steps for the product by way of a flow chart.

Fig. 1

